



**Calhoun: The NPS Institutional Archive**  
**DSpace Repository**

---

Faculty and Researchers

Faculty and Researchers' Publications

---

2014-10-02

## Mechanical Response of Self-Ion Irradiated, Single Crystal, FCC Micropillars

Hattar, K.; Zhao, X.; He, M.-R.; Sharon, J.A.; Brewer, L.N.;  
Boyce, B.L.; Gianola, D.S.

Sandia National Laboratories

---

Hattar, Khalid Mikhel, et al. Mechanical Response of Self-Ion Irradiated Single  
Crystal FCC Micropillars. No. SAND2014-17695PE. Sandia National Lab.(SNL-NM),  
Albuquerque, NM (United States), 2014.

<http://hdl.handle.net/10945/62865>

---

This publication is a work of the U.S. Government as defined in Title 17, United  
States Code, Section 101. Copyright protection is not available for this work in the  
United States.

*Downloaded from NPS Archive: Calhoun*



Calhoun is the Naval Postgraduate School's public access digital repository for  
research materials and institutional publications created by the NPS community.  
Calhoun is named for Professor of Mathematics Guy K. Calhoun, NPS's first  
appointed -- and published -- scholarly author.

**Dudley Knox Library / Naval Postgraduate School**  
**411 Dyer Road / 1 University Circle**  
**Monterey, California USA 93943**

<http://www.nps.edu/library>

# Mechanical Response of Self-Ion Irradiated, Single Crystal, FCC Micropillars

SAND2014-17695PE

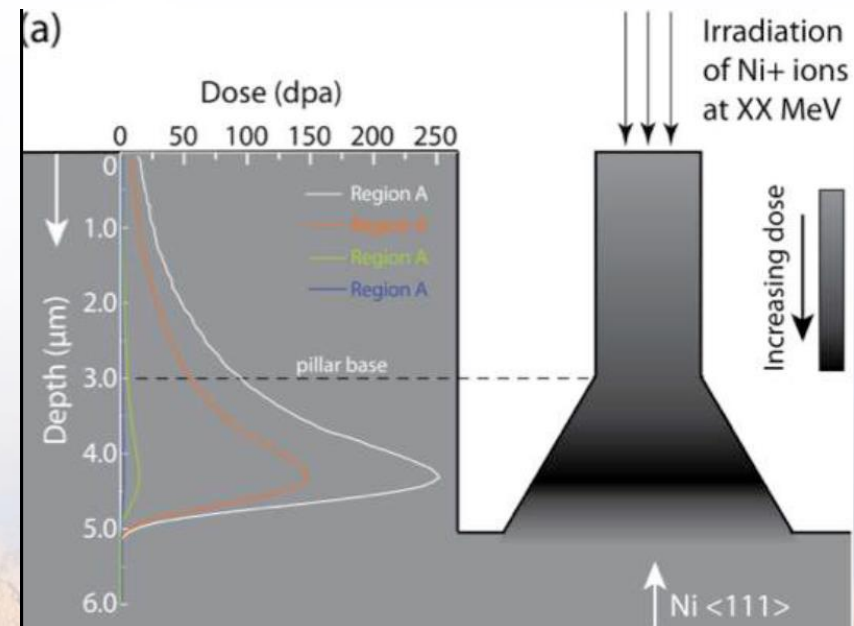
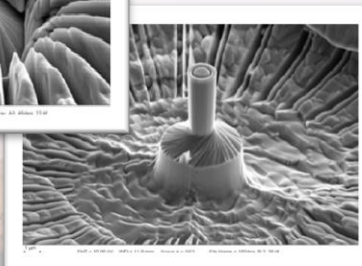
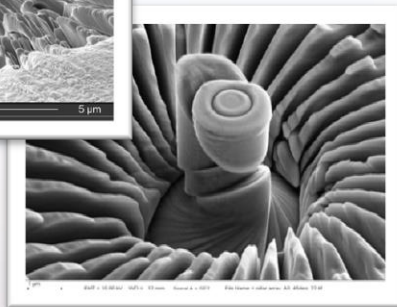
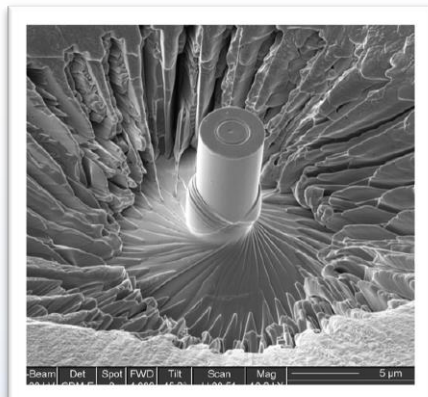
K. Hattar<sup>1</sup>, X. Zhao<sup>2</sup>, M.-R. He<sup>2</sup>, J.A. Sharon<sup>1</sup>,  
L.N. Brewer<sup>1,3</sup>, B.L. Boyce<sup>1</sup>, and D.S. Gianola<sup>2</sup>

<sup>1</sup> Sandia National Laboratories

<sup>2</sup> Department of Materials Science and Engineering, University of Pennsylvania

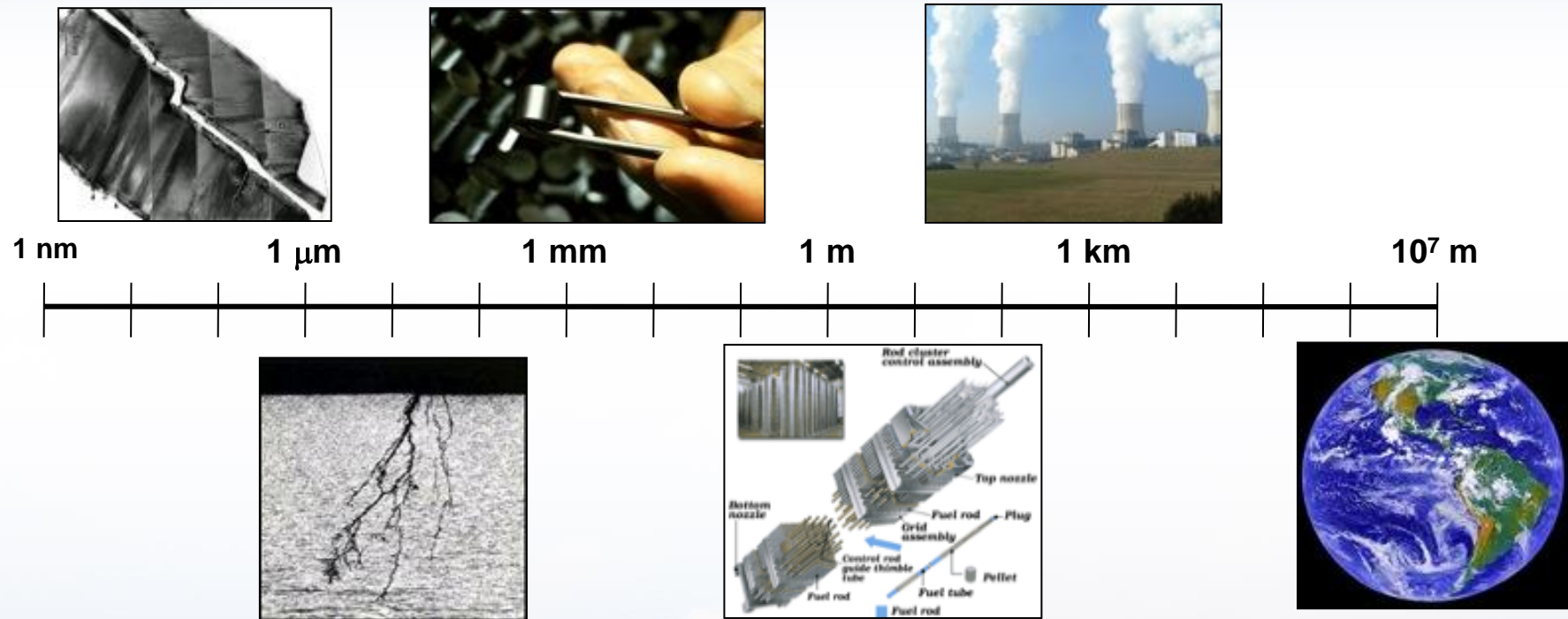
<sup>3</sup> Naval Postgraduate School

October 2, 2014



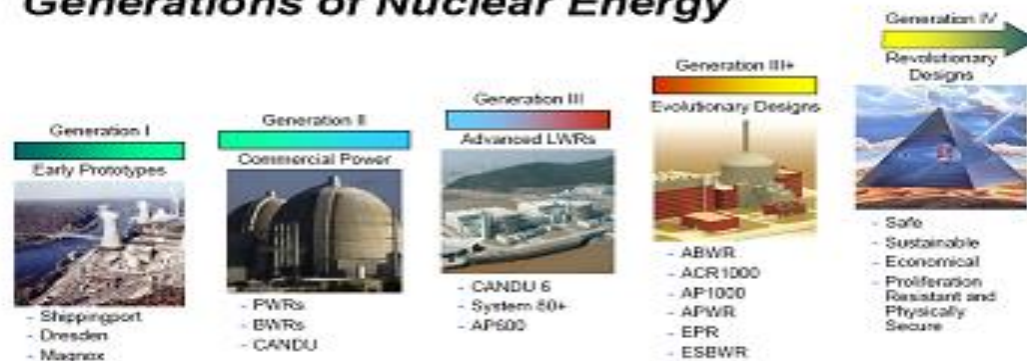
Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. SAND2009-2801P

# Investigating the **nm** Scale to Understand the **km** Scale Response of Materials in the Extremes



- Advanced Materials are Needed
- Several Theories exist for the desired microstructure
- New materials have been made
- Current Neutron fluxes require decades for testing

## Generations of Nuclear Energy



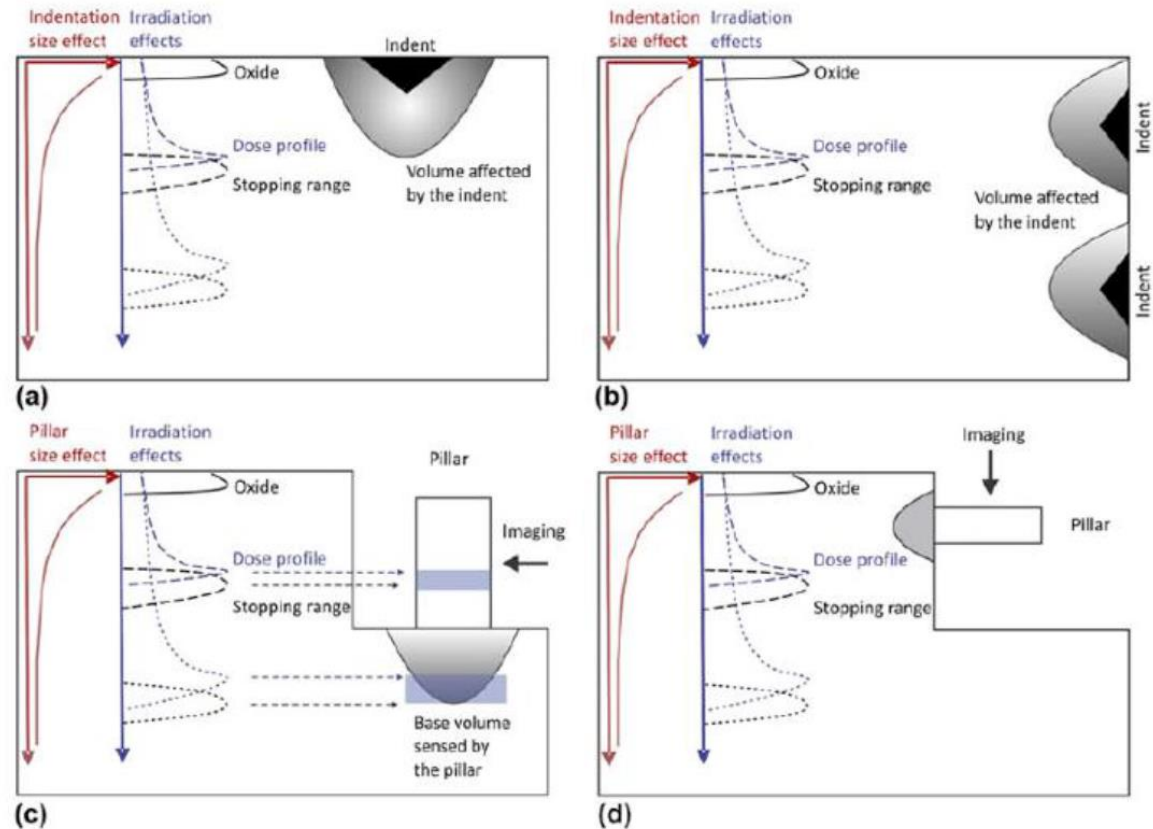
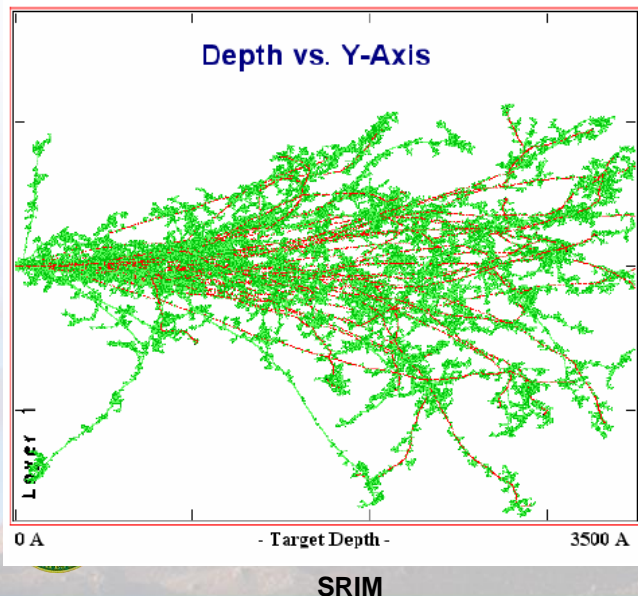
# Length Scale Limitations due to Ion Irradiation

## Advantages

- High total damage in short periods of time
- Relatively accessible

## Disadvantages

- Unknown effect of damage rate
- Limited to small volumes
- Heterogeneous microstructure

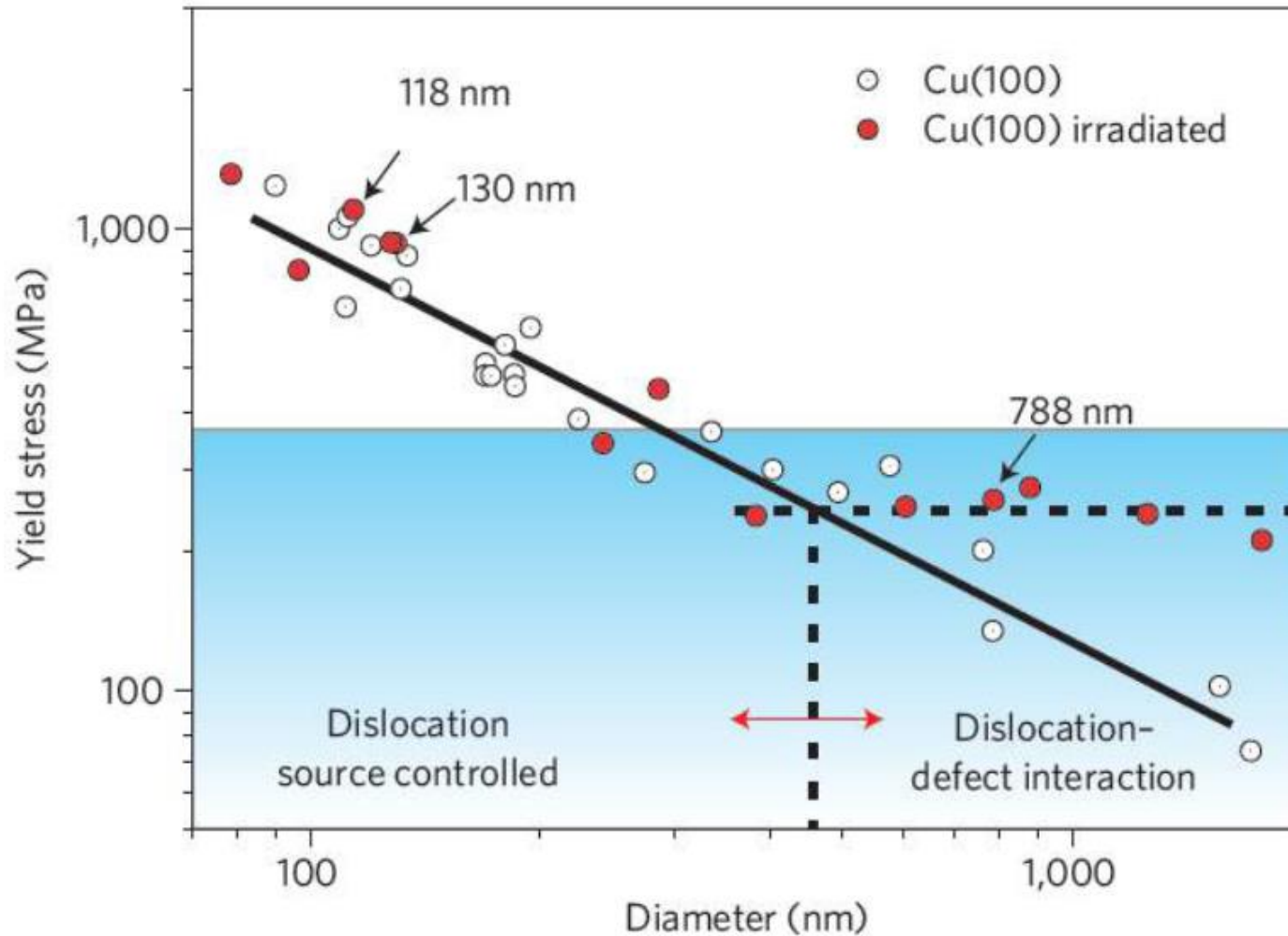


Kiener et al. JMR (2012)

Key point



# How can we decouple size effects from irradiation-induced effects



The increase in yield strength associated with the decreasing of pillar size below ~500 nm makes it difficult to identify the effects of radiation damage in small pillars.

Kiener et al. Nat. Mater. (2011)



Sandia National Laboratories

# Micropillar Compression Experiments

## Sample Preparation:

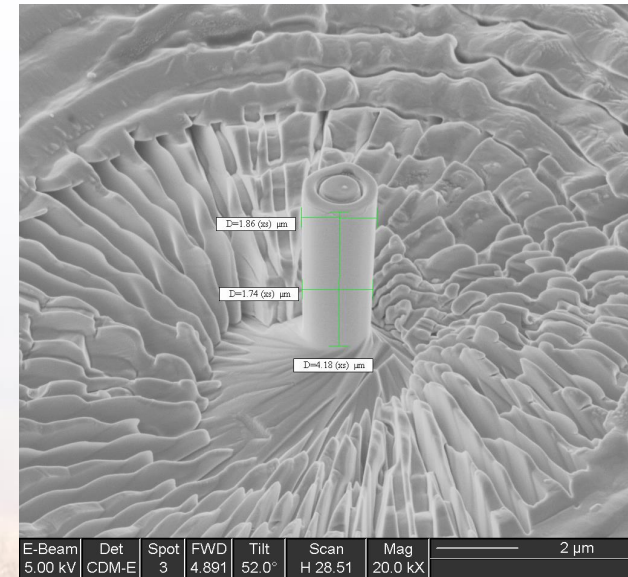
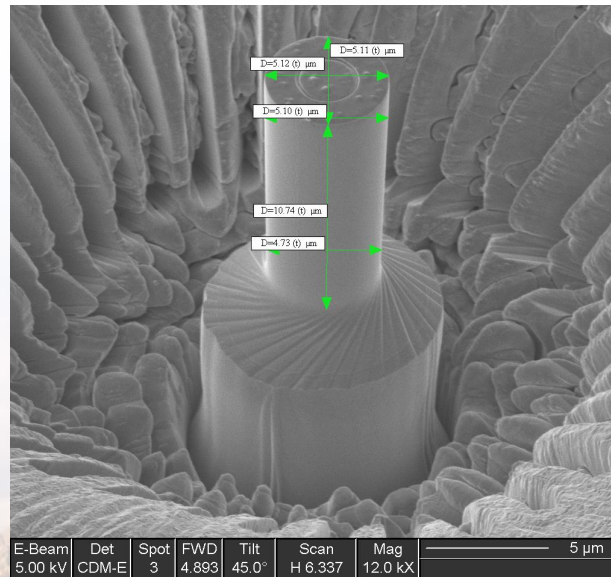
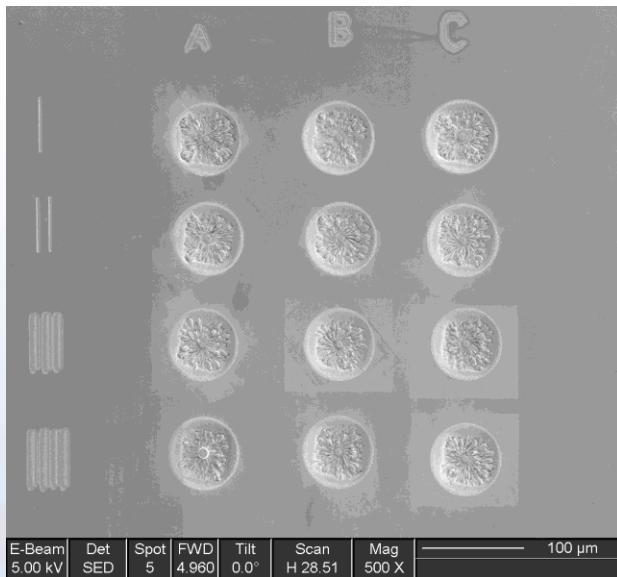
- Copper single crystals (FCC)
- Different crystallographic orientations: (100), (110), and (111)
- Self-ion Implants at 30 MeV to
- 0 (control), 50 dpa, and 100 dpa.

## Pillar Manufacturing:

- We employ Uchic's FIB lathe machining process for straight-walled cylinders.
- Array of at least 9 nominally identical pillars tested per condition to assess statistical variability.
- Height varies from 4  $\mu\text{m}$  to 10  $\mu\text{m}$

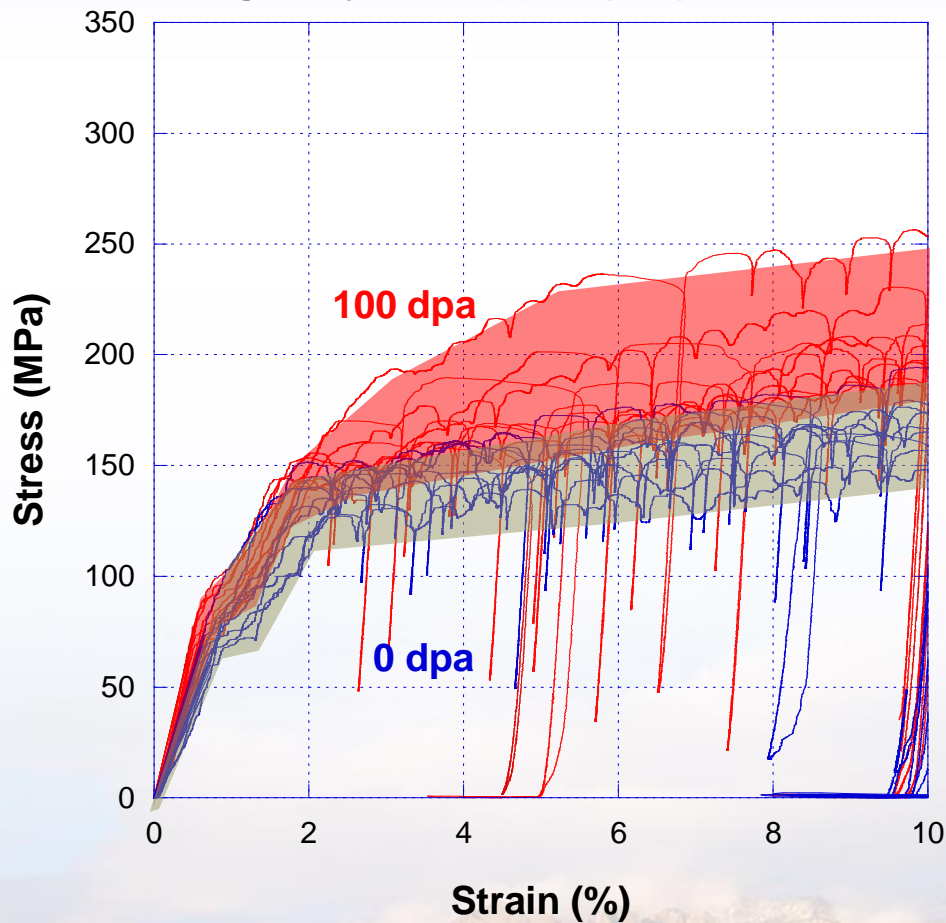
## Compression Testing:

- Hysitron Performech Nanoindenter permits  $<1$  nm and  $<1$   $\mu\text{N}$  resolution.
- 25  $\mu\text{m}$  flat ended cone indenter in feedback displacement control, rather than typical force control.
- Pillars compressed 10% strain at a strain rate of  $0.025$   $\text{s}^{-1}$ .

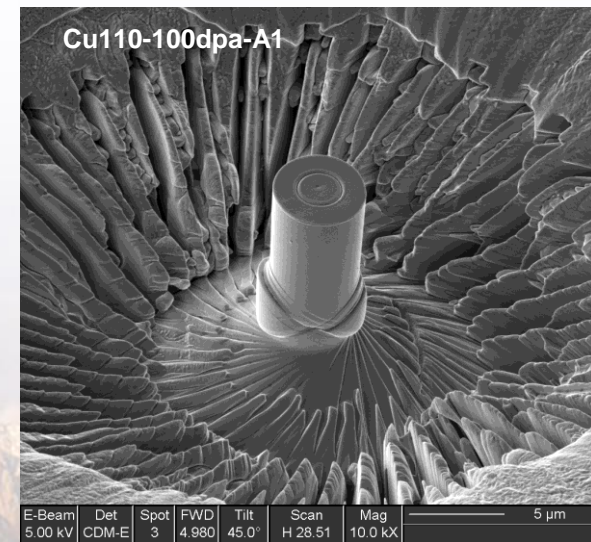
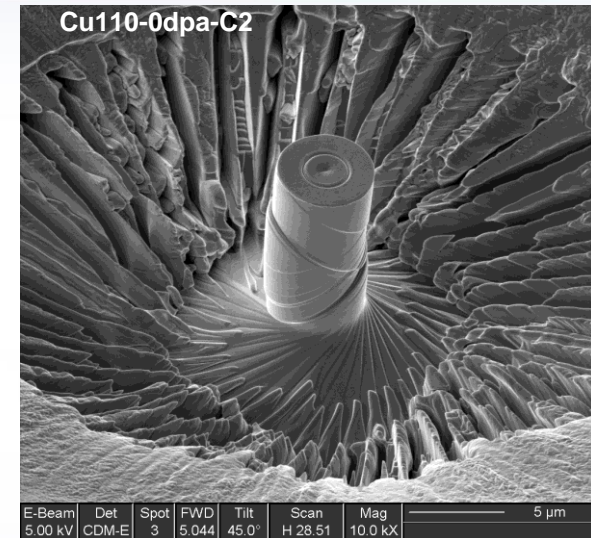


# Large Micropillar Compression

Single Crystal Copper, (110) Orientation

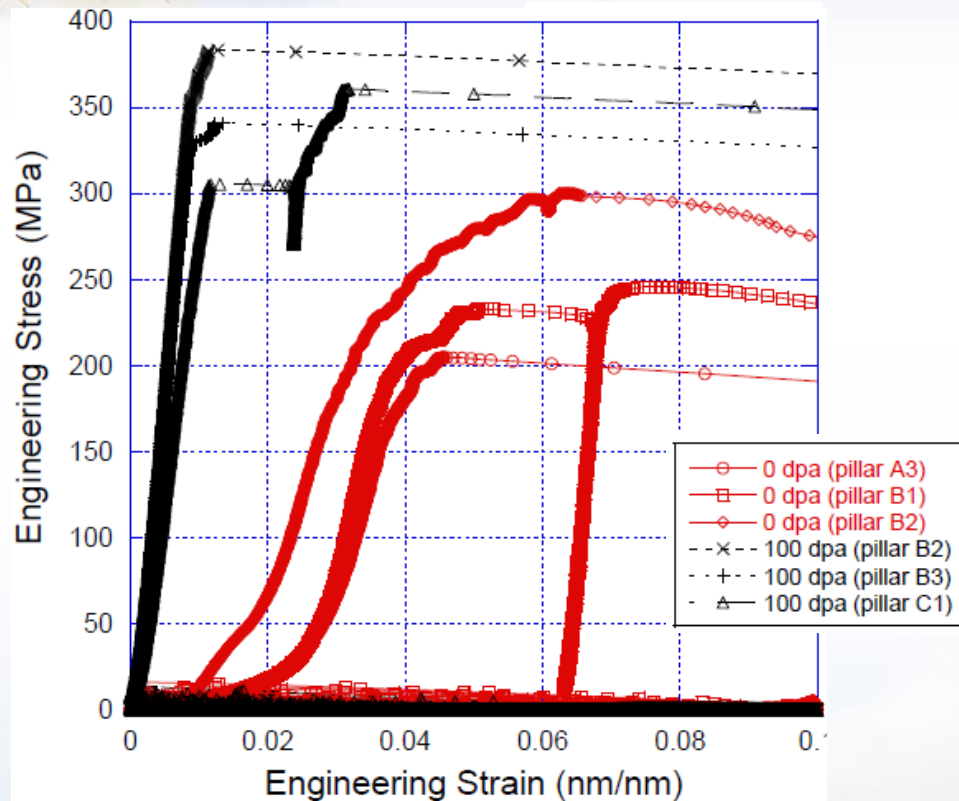


Minimal difference between the control and irradiated 10  $\mu\text{m}$ -tall pillars. Slip occurred in the bottom fraction of the pillars.

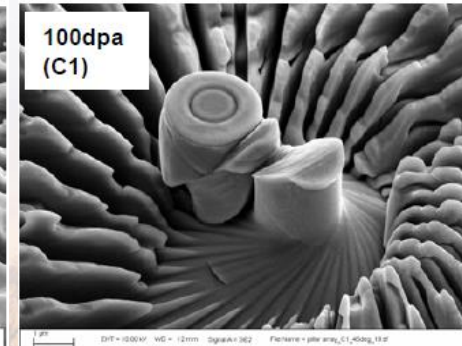
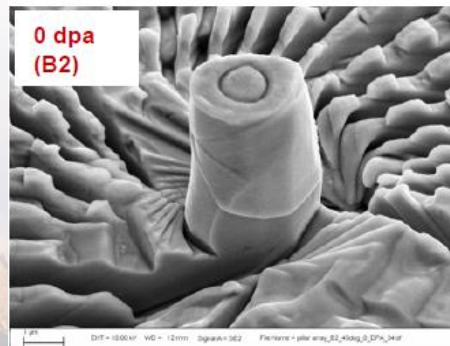
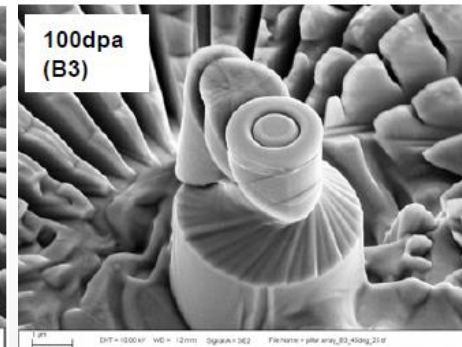
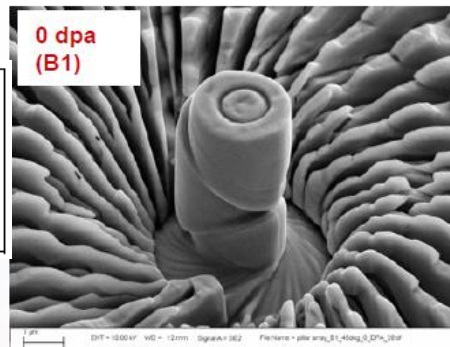
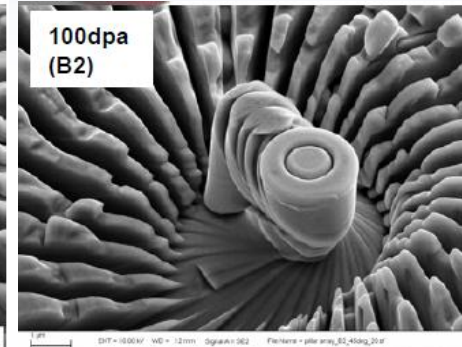
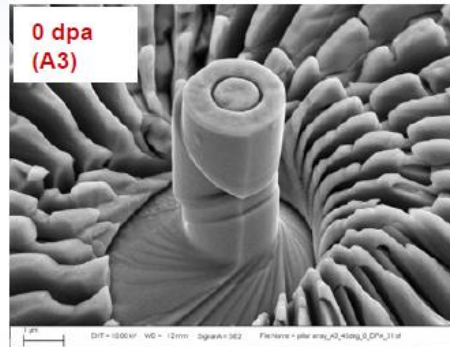




# Intermediate Micropillar Compression



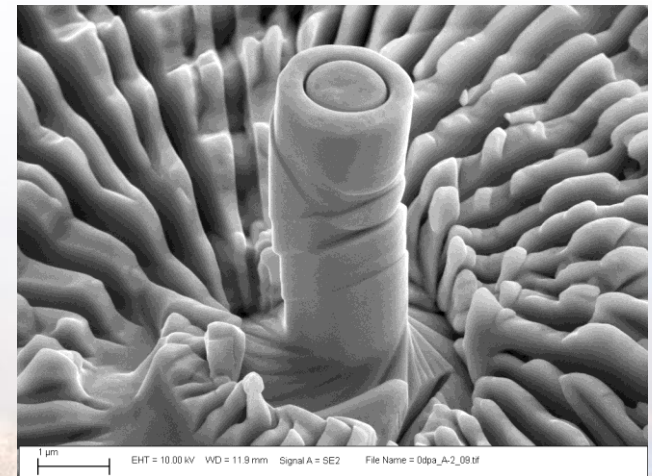
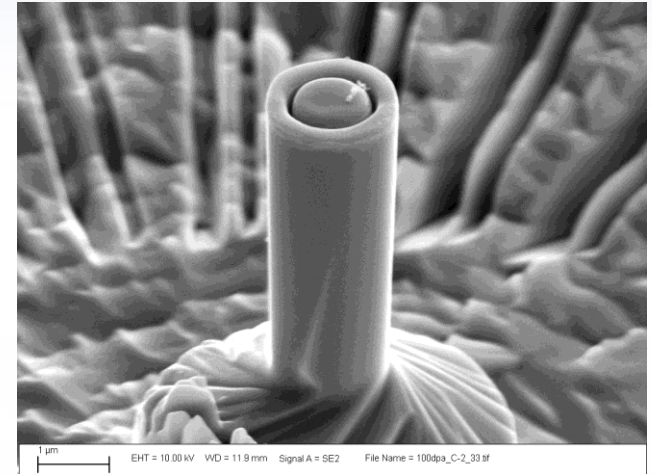
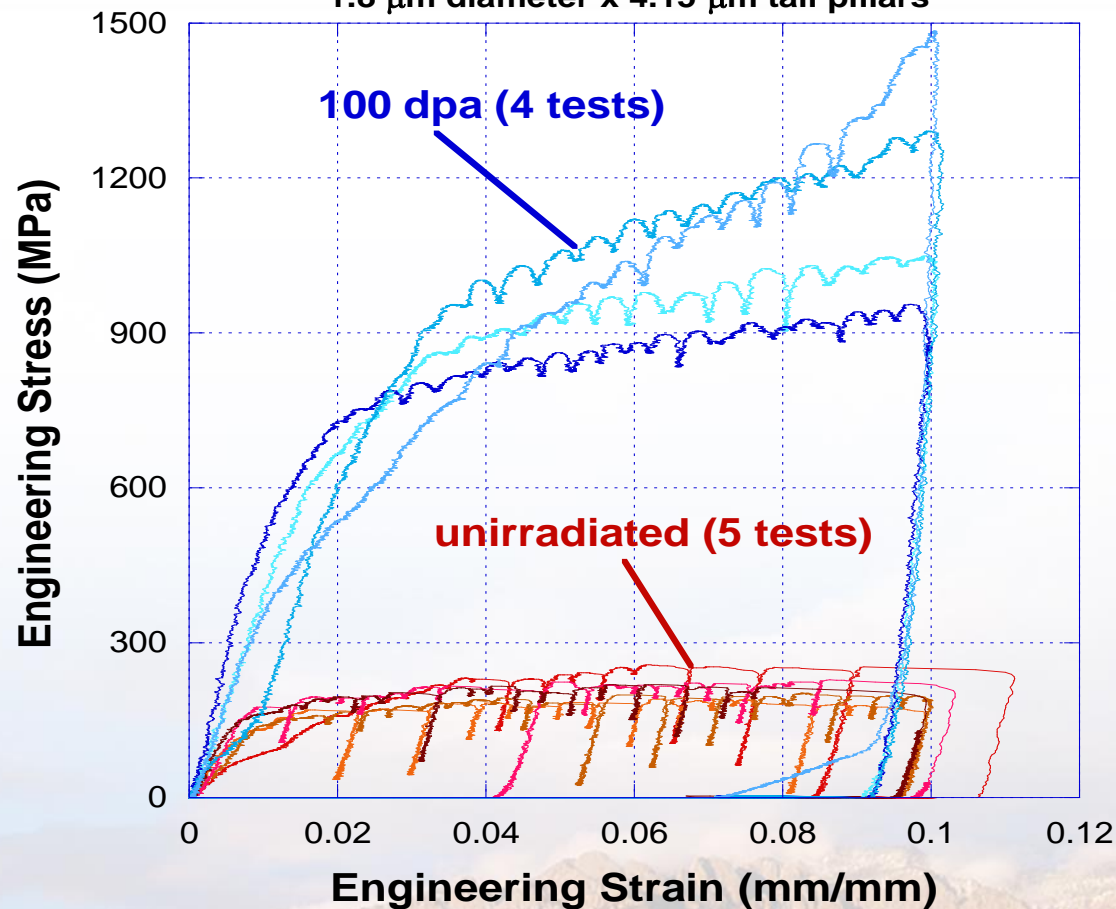
**5  $\mu\text{m}$ -tall pillars show greater distinction with catastrophic failure**





# Small Micropillar Compression

Single Crystal Cu - (110) orientation  
1.8  $\mu\text{m}$  diameter x 4.15  $\mu\text{m}$  tall pillars

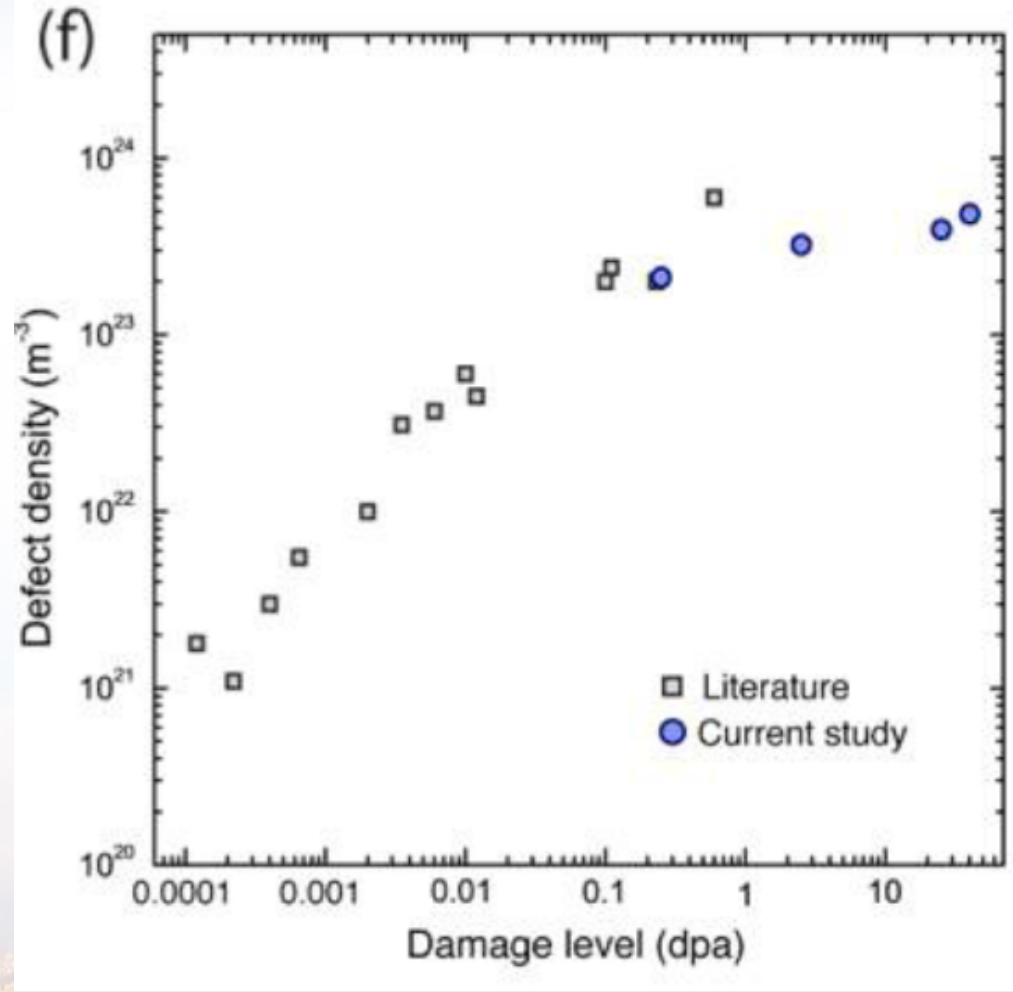
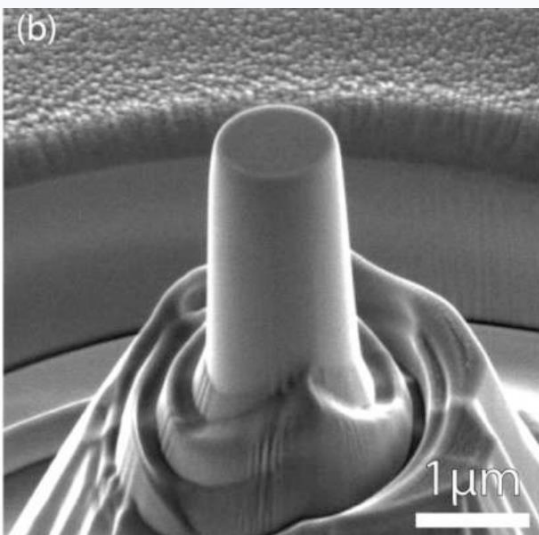
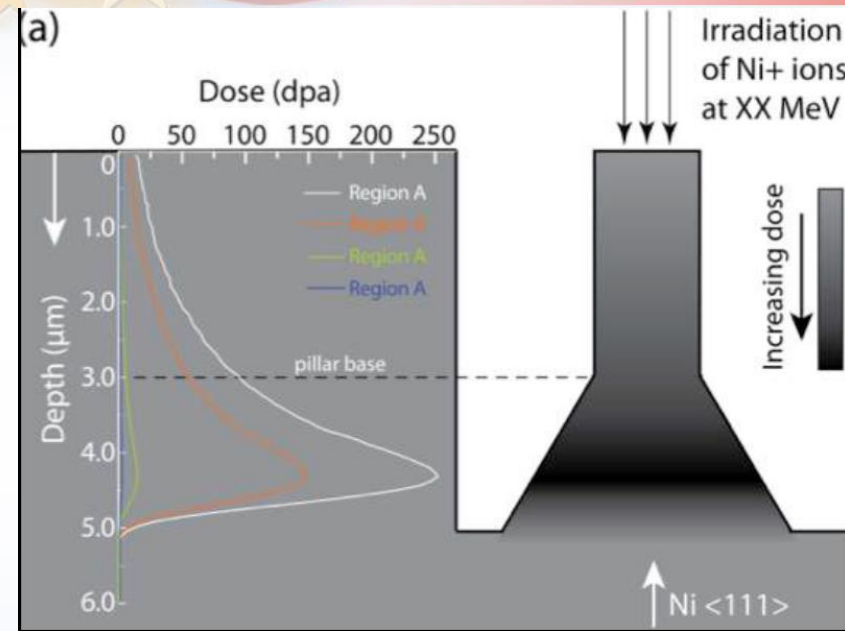


Initial tests indicate that the 4  $\mu\text{m}$ -tall pillars are 5 times stronger and show no signs of slip band formation



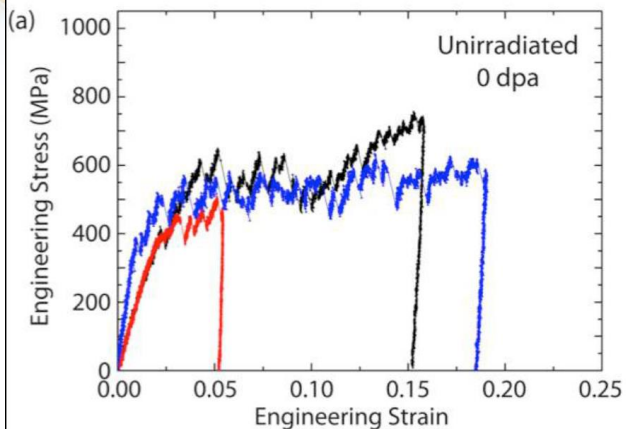
Sandia National Laboratories

# Ni implantation of $\langle 111 \rangle$ Ni



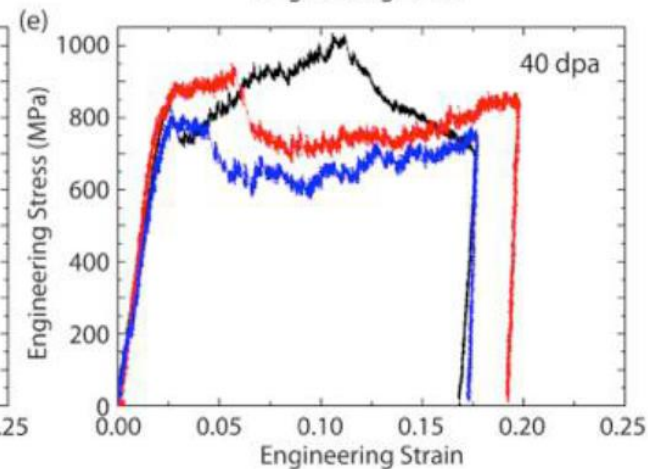
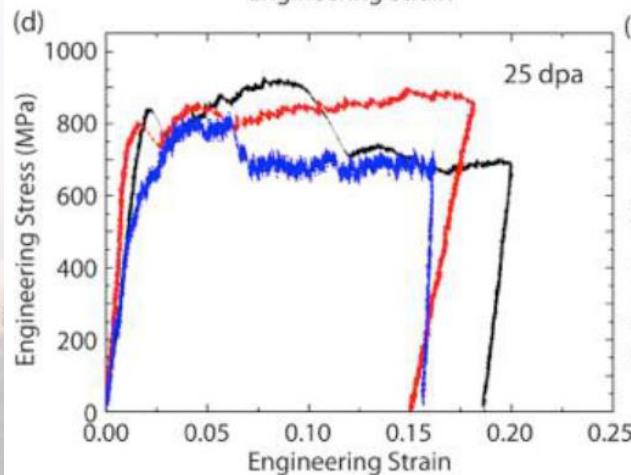
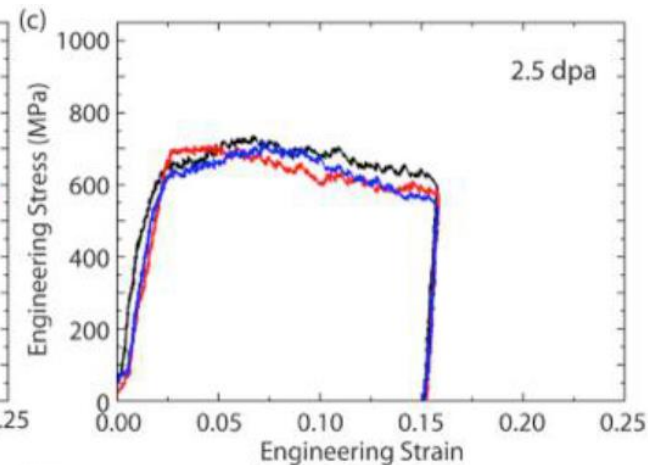
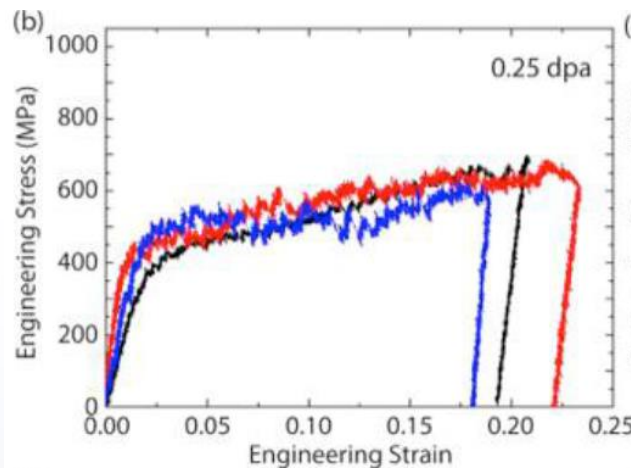
We can create similar Ni pillars and through SRIM modeling and TEM characterization, we can tailor and verify the defect structure.

# Comparison of the Mechanical Response as a Function of Dose



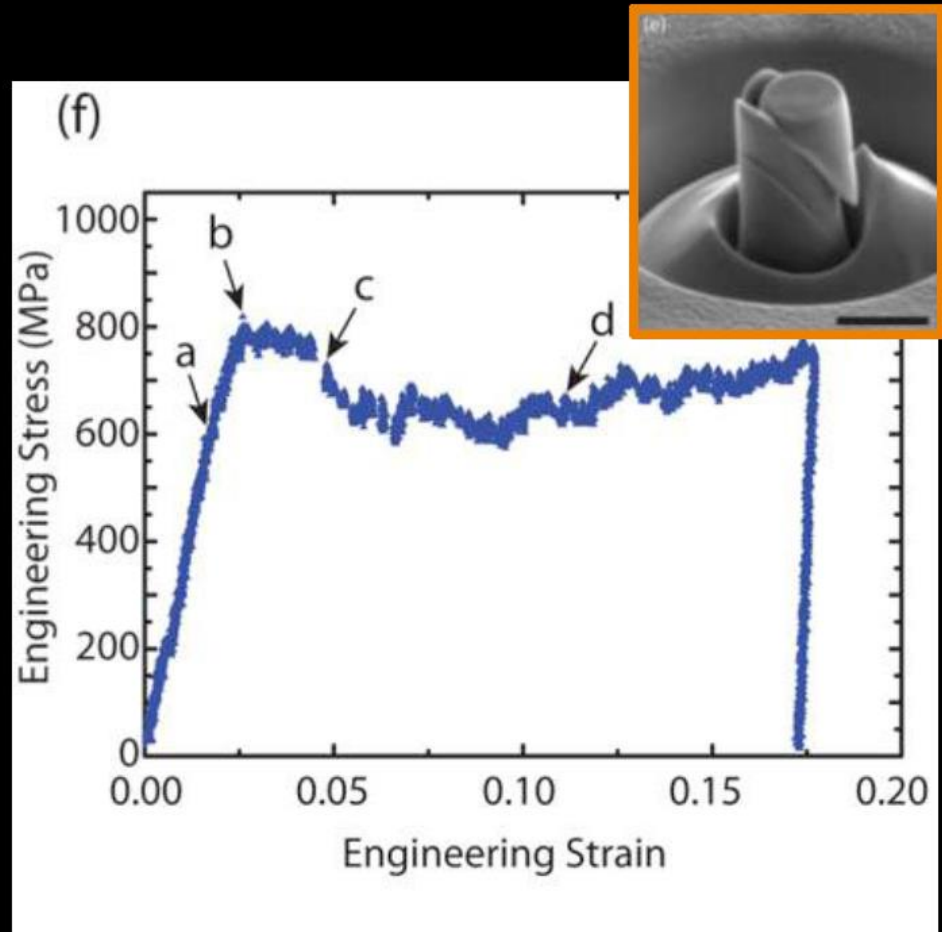
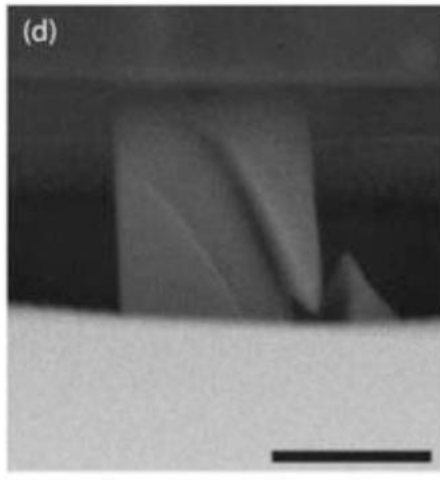
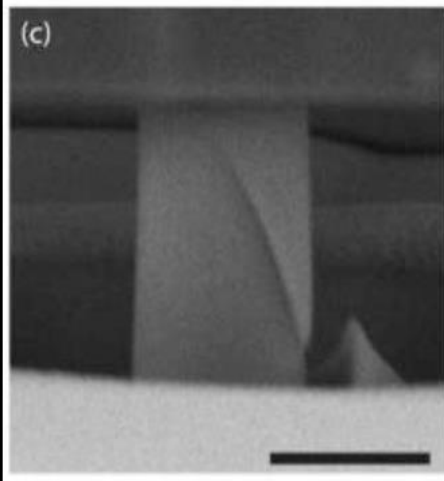
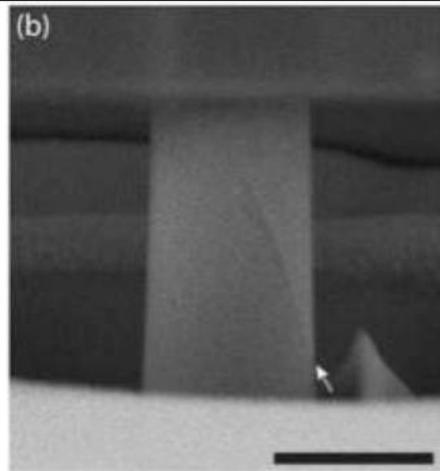
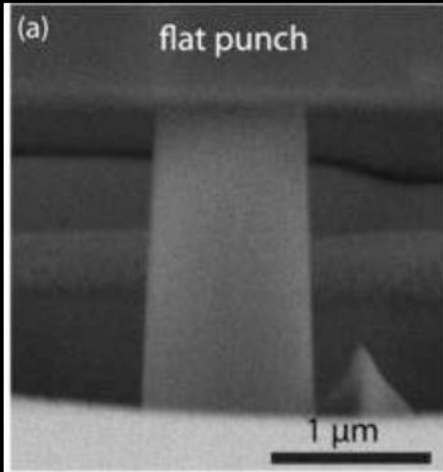
1) Strength increases with increasing damage

2) Intermittency changes dramatically and non-monotonically

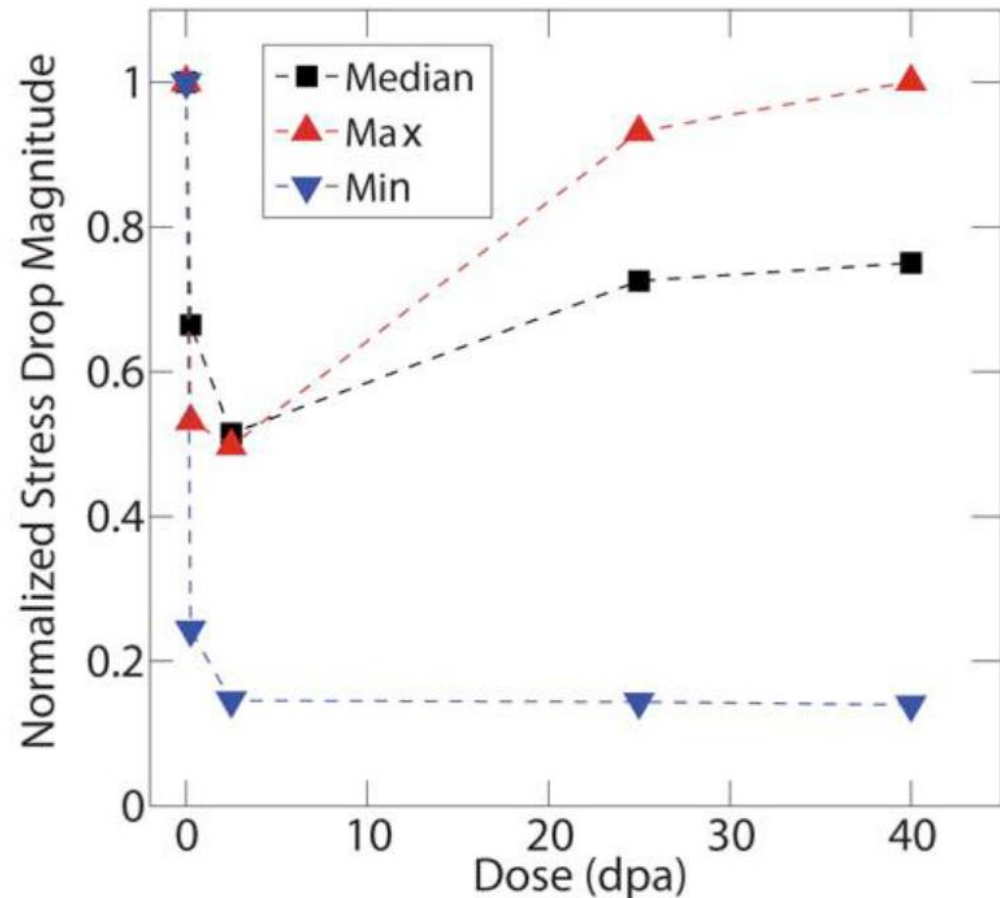
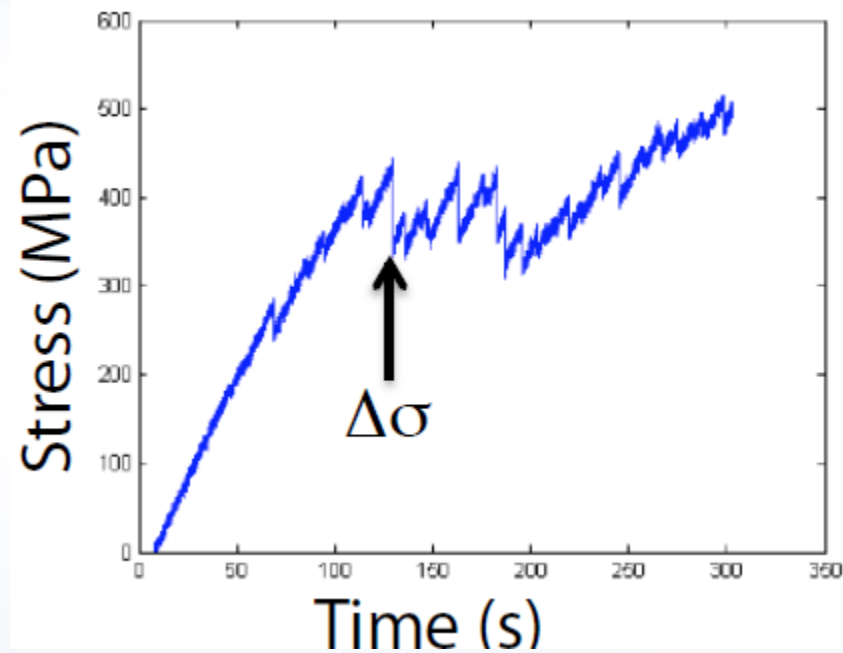




# In situ SEM Permits Correlation between Physical Slip and Intermittency in Stress-Strain Curve



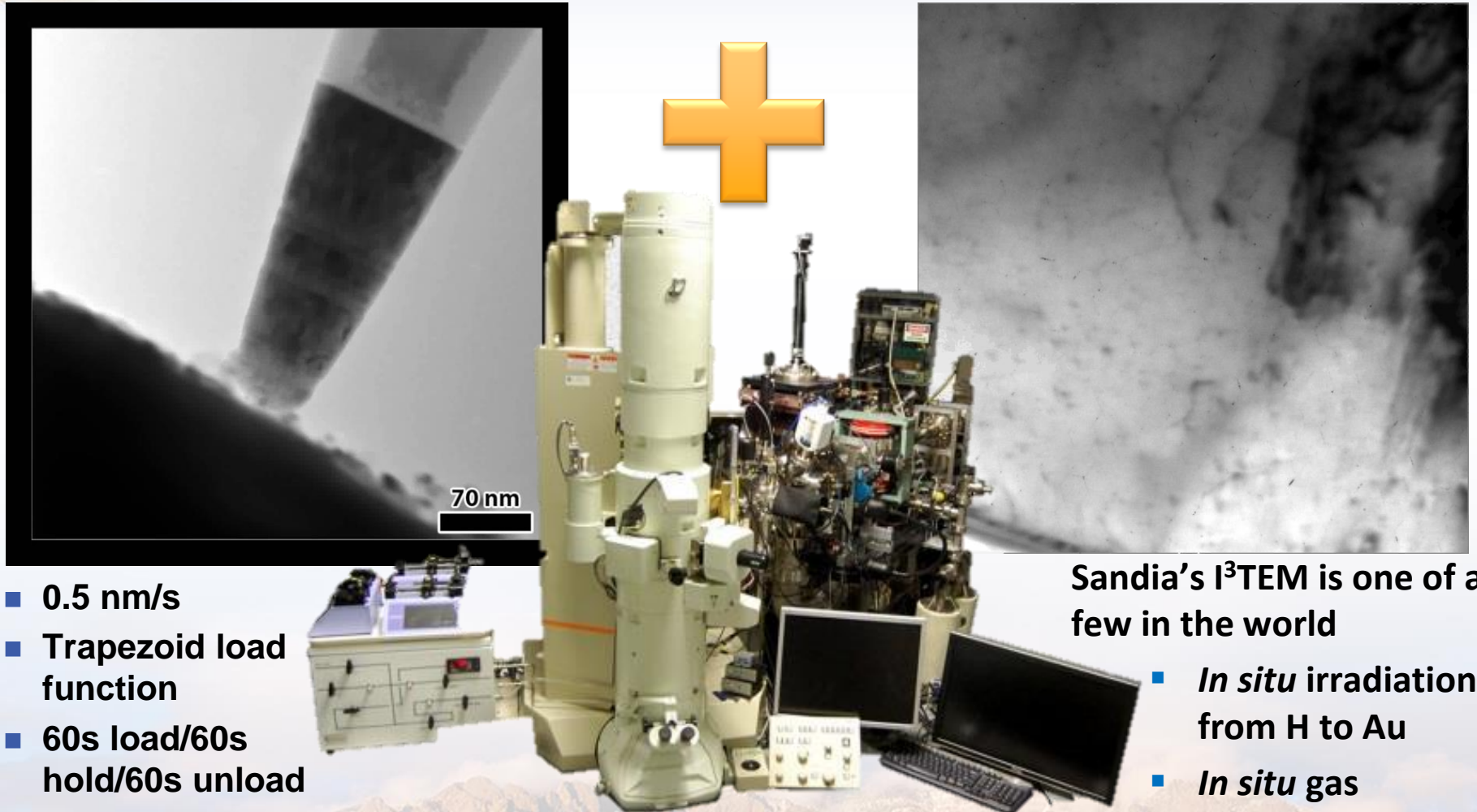
# Quantification of Stress Drops



- Minimum stress drops monotonically decreases with increasing dose.
- Maximum stress drops non-monotonically with increasing dose
- Provides insight into the role of dislocation-free channels on the mechanical response

# Future Direction

Collaborator: D.L. Buller, J.A. Scott, D.C. Bufford & W.M. Mook



- 0.5 nm/s
- Trapezoid load function
- 60s load/60s hold/60s unload

Sandia's I<sup>3</sup>TEM is one of a few in the world

- *In situ* irradiation from H to Au
- *In situ* gas implantation

**Create single crystal pillars and study the results of irradiation damage on mechanical properties and potential radiation induced creep**



Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. SAND2009-2801P



Sandia National Laboratories